

National Bureau of Standards

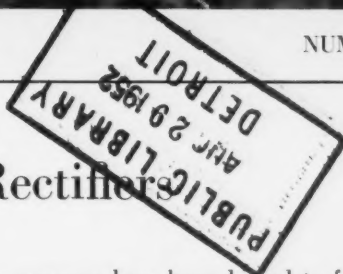
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Titanium-Dioxide Rectifiers



A NEW TYPE of rectifier recently developed by the National Bureau of Standards promises to be the first major improvement in metal-oxide rectifiers since their introduction in 1926. The new rectifier is composed of a layer of semiconducting titanium dioxide, a sheet of titanium metal, and a counterelectrode of some other conducting metal. Although the development is still in an early stage, preliminary investigations have shown that the units withstand voltage in the reverse direction reasonably well and that their properties are satisfactory at elevated temperatures. Both the initial development and subsequent detailed exploratory investigations are the work of R. G. Breckenridge and W. R. Hosler of the NBS Solid State Physics Laboratory.

The earliest practical type of metallic rectifier was the copper-cuprous oxide rectifier, invented in 1926 by Grondahl, which consists of a copper sheet covered with a layer of cuprous oxide, and a counterelectrode on top of the oxide layer. Many extensive investigations have been undertaken to develop similar rectifiers, but until now no other metal-oxide film combinations have been found that were feasible, although several nonoxide types have been produced—e. g., the selenium and magnesium-copper sulfide rectifier.

The new NBS rectifiers are prepared by forming a layer of titanium dioxide on a sheet of titanium metal and then applying a counterelectrode to the oxide sur-

face. Two processes have been devised to form the oxide layer. The first process involves heating the titanium metal first in oxygen gas and then in hydrogen gas. The other—an improved version of the first—consists in heating the titanium metal in steam at elevated temperatures.

In the first process, 1/2-inch squares of commercial titanium metal sheet, 0.020 in. thick, are polished and heated in oxygen gas to form the coat of titanium dioxide. A thin, tightly adherent coat of oxide is obtained after a treatment at 800° C for about 2 hours. The oxide layer formed in this manner becomes a very poor conductor of electricity; it is made into a semiconductor with a much greater conductivity by heating in hydrogen gas, which causes a loss of oxygen. A study of the reaction showed that heating in one atmosphere of hydrogen at 500° C for 15 minutes would produce sufficient conductivity.

After the hydrogen treatment, a counterelectrode is electroplated on the desired rectifying surface of the oxide by a-c plating techniques. A number of metals have been tried successfully as counterelectrodes, including silver, gold, tin, nickel, copper, zinc, and cadmium, although silver has been used for the majority of the work. When the current is measured for various applied voltages, it is found to flow much more readily in one direction than the other; hence, the combination acts as a rectifier.

Rectifiers produced by this process are electrically satisfactory; however, the two-step process is difficult to control and, in addition, the oxygen and hydrogen gas tends to dissolve in the heated titanium metal and make it brittle. The second process of oxide formation, using water vapor, produces the semiconducting oxide layer on the metallic titanium in one step. The most satisfactory films are formed by heating similar titanium plates in steam at 600° C for about 3 hours. The counterelectrodes are then electroplated as before to form a finished unit. This process produces a rectifier with improved electrical properties and lacks the undesirable embrittlement accompanying the other method.

While the titanium-dioxide rectifiers are still in a very early stage of development, some of their properties have aroused considerable interest. The units can withstand a reverse voltage of about 20 volts per plate. In addition, they have good properties at elevated temperatures; their performance is actually improved with increasing temperature up to about 150° C. Detailed studies have not been made of their operating life, but tests thus far are very encouraging.

The NBS titanium dioxide rectifiers bear a physical resemblance to the copper oxide type; however, there are fundamental differences between them. For example, the direction of easy flow of current is opposite in the two types. In the titanium dioxide rectifier, the electrons flow from the titanium metal base plate to the counterelectrode. The mechanism of conduction in the metal oxides is also different. In cuprous oxide the charge carriers are "holes" or electron vacancies in the lattice, while in titanium dioxide semiconductors the charge carriers are free electrons. Finally, the "barrier layer," or asymmetrical potential that must be surmounted by the charge carriers, apparently is located near the counterelectrode-oxide interface for the NBS type, while there is evidence that in the cuprous oxide



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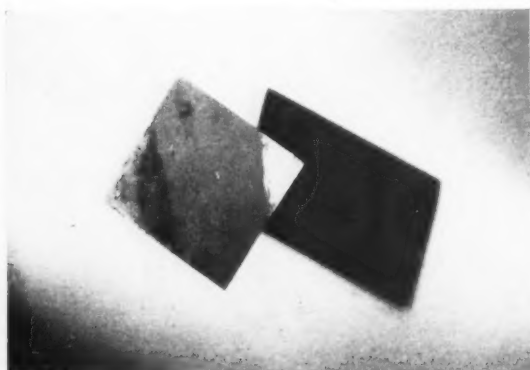
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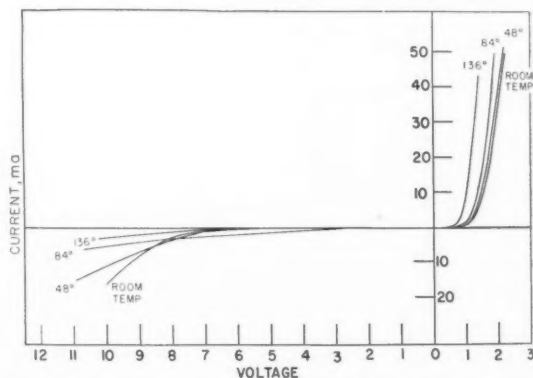
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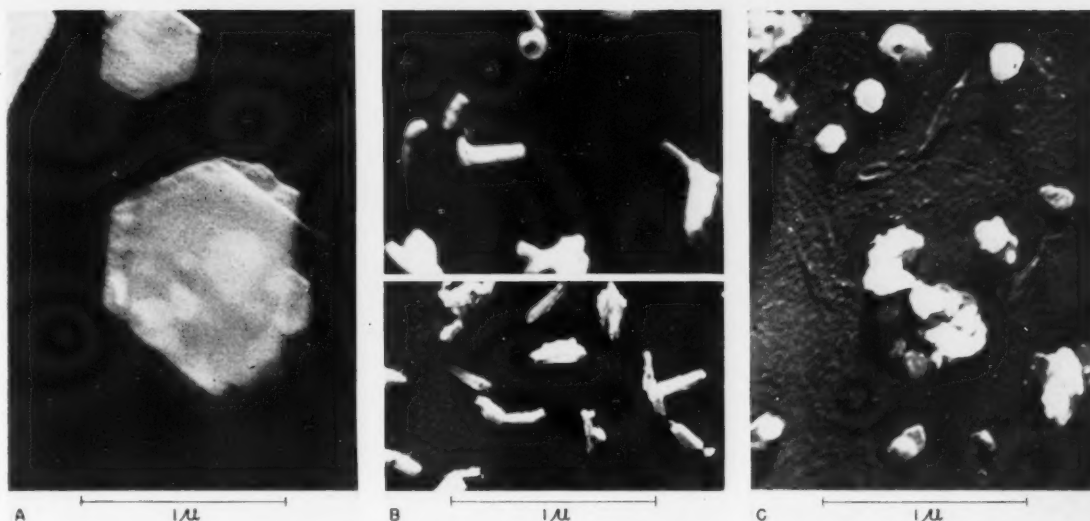
device the barrier is located at the base metal-oxide surface.

NOTE: This report is summarized from a paper presented at the Washington Meeting of the National Research Council's Conference on Electrical Insulation, October 29 to 31, 1951.



Left: laboratory test sample of the new titanium dioxide rectifier developed at the Bureau. The rectifier plate has been specially processed to show the titanium metal (light corner), the dioxide coating (center portion), and the electroplated silver counterelectrode (darker corner). The plate is made of 1/2-inch squares of commercial titanium metal sheet, 0.020 in. thick. Right: characteristics of the titanium dioxide rectifier. A graph of current (in milliamperes) vs. voltage shows the performance of the metal oxide rectifier under various operating conditions. The unit has improved characteristics at temperatures around 150° C. It is also able to withstand a voltage in the reverse direction of about 20 volts per plate.





Electron micrographs of three forms of kaolin minerals. *A*: Dry Branch, Ga., kaolinite illustrating its hexagonal-platy habit. *B*: Lawrence Co., Ind., halloysite (indianait) showing its tabular morphology. Note the unusual end view of a crystal (upper center) indicating its hollow nature (shadow length four times crystal height). *C*: Red Mediterranean soil, Beja, Portugal, exhibiting its irregular layered crystals having curved surfaces and revealing features between the pure clay mineral *A* and *B*.

Criteria for Characterizing Clays and Soils

RECENT studies by the National Bureau of Standards and the U. S. Department of Agriculture have provided data for identifying clays and soils and interpreting some of their properties. The common procedure for characterizing soil clays is based on a comparison of the results of physical and chemical determinations with the results of the same determinations on pure mineral specimens. In the present investigation three different methods—differential thermal analysis, powder X-ray diffraction analysis, and electron microscopical observations—were used to provide specific criteria for identification of kaolinite and halloysite in clays. These were evaluated for soils in which kaolin is the predominant clay mineral.

Hundreds of millions of dollars worth of clays are used annually in the United States. Fundamental studies of clays and clay technology provide vital data to ceramists, soil scientists, chemists, petroleum engineers, civil engineers, and many others. Methods of identification and investigation of the clay minerals are basic to the study of their structure and properties.

Although kaolinite and halloysite are members of the same mineralogical group (kaolin), the two are not readily differentiated because both clay minerals have the same chemical composition (approaching $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) and essentially the same basic crystal structure. The current study of these materials consisted of a correlation of such criteria as the slope ratios of the main endothermic curves obtained from graphical plots of differential thermal analysis data; the shapes, spacings, and intensities of the interplanar

reflections obtained from X-ray powder diffraction patterns; and the crystal shapes and sizes determined by electron microscopy. The results obtained by all three independent methods were entirely consistent for these two pure kaolin minerals. These specific characteristics were used for identifying the kaolin content of many red and yellow soils.

Hexagonal-shaped kaolinite was observed in many soils (fig. A) while cylindrical-shaped halloysite was present in others as a minor constituent (fig. B). However, the kaolin mineral in soils is often in the form of small particles and is poorly crystallized, with random stacking of structural layers, having some interleaving of water or the interlayer spacing distributed in other ways. Such soil kaolin has certain structural properties of halloysite according to differential thermal analysis and X-ray diffraction: it exhibits features between that of halloysite and kaolinite according to electron microscopy. It is, however, sufficiently different to represent a separate third morphological class of kaolin mineral and possibly represents an end form rather than a transitional one (fig. C). The kaolin mineral present in some soil clays is commonly associated with other minerals that act as diluents or contaminants. In such cases clues but not complete identification are provided.

For further technical details see, "Criteria for the characterization of kaolinite, halloysite, and a related mineral in clays and soils," by Luis Bramao (*Estacao Agronomica Nacional, Sacavem, Portugal*), J. G. Cady and S. B. Hendricks (*U. S. Dept. of Agriculture*), and Max Swerdlow (*NBS, Soil Sci. 73, (1952)*).

Effect of Strain-Temperature History on the Low-Temperature Properties of Ingot Iron

INSIGHT into the effect of the strain-temperature history and strain aging of specimens of ingot iron on their tensile properties at low temperatures is provided by a recent study conducted by G. W. Geil and N. L. Carwile of the NBS Metallurgy Division. Tensile specimens were extended at selected temperatures, ranging from -196° to $+100^{\circ}$ C, to specified strain values and were subsequently extended to fracture at different temperatures within the above range. By comparing the tensile data obtained in these tests with those of specimens extended to fracture in single-stage tests at the same temperatures, much valuable information on the combined effect of previous strain and temperature was obtained. This study was carried out as part of an extensive NBS investigation of the deformation of metals at low temperatures.

The manner in which metals deform, with ultimate failures or fracture depends on a number of factors. At high temperatures metals creep, i. e., extend plastically at stresses below the yield strength. At room temperatures, the different metals vary greatly in their ductility at fracture. At sub-zero temperatures, some metals and steels become noticeably embrittled, whereas other steels and certain nonferrous alloys may be tougher than at room temperature. To obtain a better understanding of the factors involved, the Bureau is conducting a long-range investigation of the flow, fracture, and ductility characteristics of metals and alloys over a wide range of temperatures.

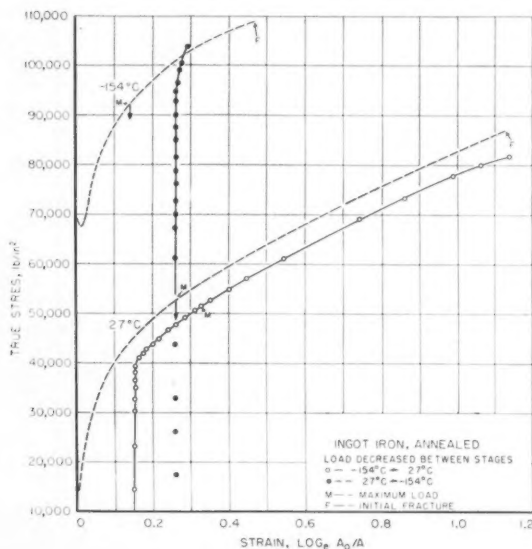
In recent years, several investigations have clearly indicated that the true¹ stress-strain relationship for metals and alloys extended in tension depends not only

on the instantaneous values of strain, strain rate, and test temperature but also on the previous strain-temperature history of the specimen. However, very little information has been available on the effect of previous strain-aging on the stress-strain relationship. The NBS study thus fills an important gap in present knowledge of the low-temperature properties of metals.

On the basis of processing and previous heat treatment, the ingot iron samples studied by NBS could be classified as (1) annealed, (2) hot-rolled, (3) quenched and tempered, (4) normalized, or (5) cold-drawn. They were all machined into cylindrical tensile specimens and were extended in tension to specified strain values in a pendulum-type hydraulic testing machine of 50,000 pounds capacity. While undergoing deformation, the specimens were kept completely submerged in a liquid bath maintained at the desired temperature, except in those tests made at room temperature. Through use of a specially designed reduction-in-area gage² developed at NBS in 1949 for low-temperature application, the diameter of each specimen was measured during extension to an accuracy of ± 0.0001 inch.

The specimens were first extended at selected temperatures ranging from -196° to $+100^{\circ}$ C. After an interval of about one-half hour, they were again extended—this time to fracture—at a different temperature within the same range. During the interval between the two stages of each test, the specimen was maintained at the lower temperature in order to minimize any strain aging of the iron during this period. Simultaneous load and diameter measurements were made throughout the course of each stage of deformation, and true stress-strain curves were plotted from these data. The deviation of the true stress-strain curve for the second stage of the tension tests from the true stress-strain curve for a single-stage test at the same temperature as that used in the second stage gave a measure of the effect of the strain-temperature history during the first stage of the test on the tensile properties of the metal.

For ingot-iron specimens extended in tension in the temperature range studied, the total work hardening (increase in true stress with true strain) is mainly the resultant of the two processes known as strain hardening and strain aging. Strain hardening is caused by crystal deformation and increases as the testing tem-



True stress-strain values for annealed ingot iron specimens extended in tension at NBS in two-stage tests. The closed circles represent values obtained during the extension at -154° C of a specimen that had been previously extended to the maximum load (M) at 27° C. The open circles represent values obtained during the extension at 27° C of a specimen that had been previously extended to the maximum load at -154° C. The broken-line curves show the true stress-strain relationship at the indicated temperatures for specimens extended to fracture in a single-stage test.

An ingot iron specimen, immersed in a bath of liquid nitrogen, is tested in tension at NBS. The Bureau is conducting a general investigation of the mechanical properties of metals at low temperatures.

perature is lowered. Strain aging, on the other hand, is generally associated with the precipitation of an element from solid solution and increases within certain limits with rise in temperature. The work hardening of ingot iron during deformation in tension at temperatures below -120°C is mainly strain hardening; thus at these temperatures the total work hardening increases with lowering of the temperature. However, as the testing temperature is raised above -120°C , the strain aging during the deformation of the specimen increases sufficiently to become a dominating factor, and the work hardening then increases with increase in testing temperature.

The relative proportions of strain aging and strain hardening at the various temperatures for the five conditions of the ingot iron were determined on the basis of the known tendencies of these two types of work hardening to increase or decrease with temperature. Until now the general opinion has been that strain aging of ingot iron is insignificant at subzero temperatures. However, the data obtained by the Bureau show that strain aging has a pronounced effect on the true stress-strain relationship of ingot iron specimens extended in tension at temperatures as low as -120°C .

The results of the study also shed new light on the so-called "rheotropic" brittleness of ingot iron. Some investigators have recently reported that a part of the brittleness in steels and other metals at low temperatures can be removed by cold work at temperatures above that of the transition range from ductile to brittle behavior. Such brittleness has been termed rheotropic, and a large part of the deficiency in ductility of annealed steels and some other metals at low temperatures has been reported to be rheotropic.

The data obtained in the NBS investigation show that the rheotropic brittleness of ingot iron depends upon the initial condition of the specimen. For example, it was found that cold working of hot-rolled or normalized specimens by extension in tension at room



temperature removed some of their brittleness at low temperatures, whereas similar cold working of annealed specimens increased their low-temperature brittleness.

For further technical details, see Effect of strain-temperature history on the flow and fracture of ingot iron at low temperatures, by G. W. Geil and N. L. Carwile, *J. Research NBS* **48**, 349 (1952) RP 2329. See also Tensile properties of ingot iron at low temperatures, by G. W. Geil and N. L. Carwile, *J. Research NBS* **45**, 129 (1950) RP 2119.

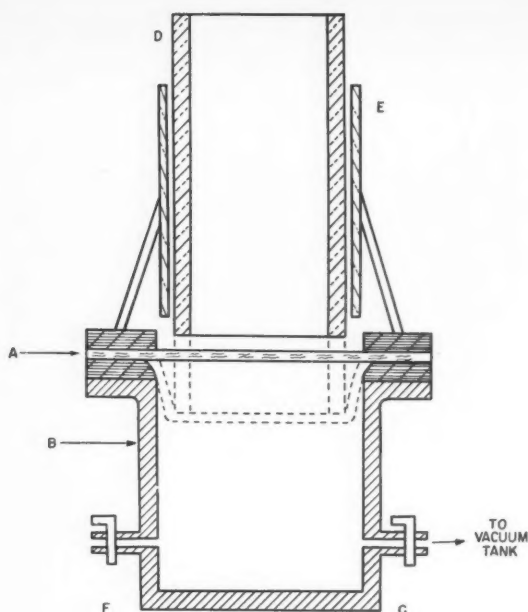
¹In computing "true stress," the reduction in cross-sectional area of the specimen during extension is taken into account.

²A reduction-of-area gage for low-temperature application, *NBS Tech. News Bull.* **34**, 24 (Feb. 1950).

Improvement of Acrylic Aircraft Glazing by Stretch-Forming

ACRYLIC plastic glazing has been used for many years in astrodomes, gun turrets, antenna covers, and other transparent curved enclosures in military aircraft. However, in spite of its good weathering and optical properties and the ease with which it can be formed into shapes with compound curvature, this material has several disadvantages such as low impact strength and craze cracking. Perhaps the most serious of these is its tendency to craze under stress, especially in the presence of organic solvents. This results not only in reduced visibility but also in lowered resistance to both steady and impact loads.

Until now, little information has been available concerning the effect of forming on the crazing and strength properties of the acrylic plastic sheet. To provide the necessary data, B. M. Axilrod and associates of the Bureau's plastics laboratory recently made a study of the effects of moderate biaxial stretch-forming on acrylic plastic glazing. Results of their investigation, which was sponsored by the National Advisory Committee for Aeronautics, indicate that suitable prestretching may provide an effective means for improving the crazing resistance of the material.



Measurements on a typical astrodome had shown that the material at the apex is biaxially stretched in the forming process about 40 to 50 percent in the plane of the sheet of material; the initial NBS studies were therefore made on samples stretched approximately this amount. To obtain a flat sample uniformly stretched in two dimensions, a sheet of the plastic was first heated to a temperature in the rubbery range for the material (120° and 140° C for the general-purpose and heat-resistant grades, respectively). The sheet was then clamped to the open end of a cylindrical forming vessel, and a partial vacuum was produced quickly in the vessel to force the sheet down into the chamber. An open-ended cylindrical tube, or form, was inserted into the plastic "bubble," and the vacuum was destroyed, causing the heated rubbery plastic sheet to retract around the form. After the plastic had become rigid, it was removed from the forming vessel; it then had the appearance of a top hat. The test specimens were taken from the flat top portion of the "top hat."

Specimens from the formed pieces as well as from the unformed portions of the same sheets were subjected to various tests, including standard tensile tests, stress-solvent crazing with benzene, long-time tensile loading, and accelerated weathering. The specimens used for the long-time tensile loading and stress-solvent crazing tests had a tapered central portion. This design resulted in a gradation of stress in the loaded specimen and made it possible in the solvent crazing tests to ob-

A sheet of aircraft glazing stretch-formed in the shape of a hat is removed from a vacuum forming vessel. The plastic sheet is still attached to the cylindrical form on which it was shaped. By this technique, a flat sample stretched uniformly in two dimensions is obtained.

Schematic diagram of the apparatus used by the Bureau in the experimental stretch-forming of aircraft glazing. A sheet of the plastic is first heated until it becomes rubbery and is then clamped at A across the top of a cylindrical forming vessel B. A partial vacuum is produced quickly in the vessel, forcing the sheet down into the chamber. When the form D is inserted into the plastic bubble and the vacuum is destroyed, the elastic plastic sheet retracts about the form, and remains stretched in the shape of a hat.

tain a threshold crazing stress by testing only a few specimens.

In the long-time tensile tests, values of the threshold stress were obtained as a function of time by observing the tapered specimen at intervals after the load was applied. The threshold stresses for the different specimens were plotted against time, and from the graph average threshold values for 1, 10, and 100 hours were obtained. As the crazing behavior of acrylic plastic sheet is known to be affected by temperature and relative humidity, the long-time tensile loading tests were performed in a controlled-atmosphere room operated at 23° C. Half of the tests were made at 50 percent relative humidity; the other half were made at about 95 percent relative humidity.

The results of the NBS experiments indicate that biaxially stretch-forming the acrylic plastic 50 percent does not affect its tensile strength or secant modulus of elasticity in tension. However, the elongation at failure is greatly increased, from approximately 10 percent to about 60 percent.

The appearance of the tensile specimens after the tests led to some interesting conclusions. First, it was

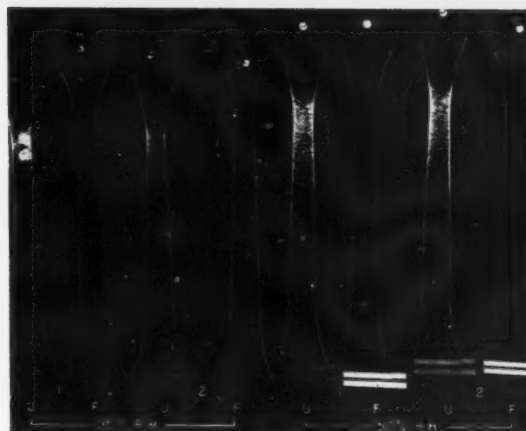


Specimens of a heat-resistant sample of acrylic plastic aircraft glazing after 7 days of tensile loading. The formed specimens (F) were subjected to a stress of 6,000 lb/in² at the minimum section while the unformed specimens (U) were subjected to a stress of 5,000 lb/in² at the same place. Note the absence of crazing in the formed specimens. The four specimens at left were tested under a relative humidity of 95 percent while those at right were stressed under a relative humidity of 50 percent.

observed that the broken tensile specimens of the formed pieces have a laminar character unlike the unformed material. Next, an examination of the fracture surface on each of the broken tensile specimens showed that in every instance there was a small mirror-like area oriented perpendicularly to the tensile load. From an examination of broken solvent-crazed specimens and other evidence, it was concluded that fracture in the acrylic material begins in the mirror area and that this area is an extension of a crazing crack.

The effect of the biaxial stretch-forming on the threshold stress for stress-solvent crazing with benzene was to increase this stress about 75 percent for both the general-purpose and heat-resistant grades. Also, there was a tendency for the crazing cracks to be somewhat finer and more closely spaced on the formed as compared to the unformed specimens.

In the long-time tensile loading tests, the threshold stresses for crazing after 100 hours of loading were about 2,700 and 3,700 pounds per square inch for unformed material of the general-purpose and heat-resistant grades, respectively. The threshold crazing stresses of all the formed samples were about 40 to 50 percent greater than the stresses for the corresponding



unformed material. Although no significant difference in threshold stress due to relative humidity was noted, the nature of the crazing at the two relative humidities was markedly different. The cracks at 95 percent relative humidity appeared finer and more closely spaced and were almost always noticeably shorter than those at the lower relative humidity.

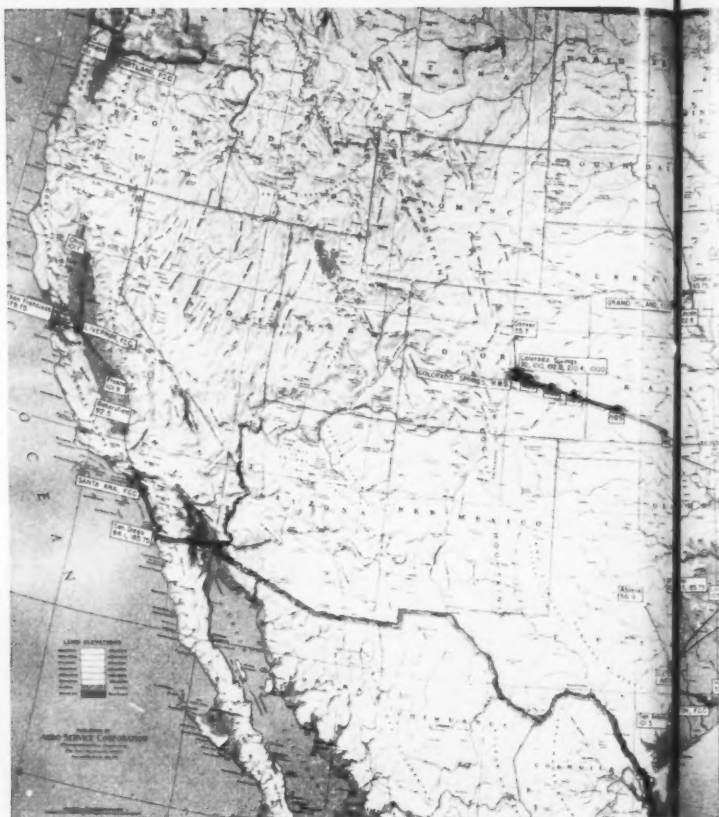
The improvement in the crazing properties of acrylic plastic sheet produced by moderate biaxial stretch-forming suggests that enclosures made from stretched sheets have superior crazing and strength properties as compared to enclosures formed with little or no stretching. As an alternative to the use of prestretched material to achieve improved craze resistance and toughness in acrylic enclosures, there is the possibility of preparing a more highly stretched enclosure directly, such as by forming a larger and more deeply drawn enclosure than required and then using only the central portion of the formed piece. Either method would improve the craze resistance, particularly at the rim, where there is likely to be stress concentration and where contact with solvents of adhesives used to seal the enclosure is quite likely; in a normally formed enclosure, the craze resistance is a minimum at the rim. The laminar structure of the stretched acrylic sheets also offers the possibility of obtaining resistance to shattering of pressurized acrylic canopies by shrapnel with the necessity of employing the heavier and more expensive acrylic laminate made with a safety-glass type of vinyl interlayer.

NOTE: A complete report of this work will be published soon as a National Advisory Committee for Aeronautics Technical Note, and a brief account will also appear in the Journal of Research of the National Bureau of Standards.

Specimens of a heat-resistant acrylic glazing material after stress-solvent crazing tests at the Bureau. Note that crazing is much more pronounced in the unformed (U) than in the stretch-formed (F) specimens. S_0 is the stress in lb/in² at the minimum cross section of the specimen, and S_{sc} is the threshold crazing stress.



Tropospheric Propagation Research



DURING World War II, the development of techniques and applications for the portion of the radio frequency spectrum above 50 megacycles was accelerated. Both experimental techniques and equipment development advanced more rapidly than basic research in propagation phenomena. New applications have included radar, communications with aircraft, navigational and guidance systems for aircraft, ground-to-ground radio relay and communications systems, and new systems of broadcasting such as frequency modulation, television, and facsimile. At the close of the war, it became apparent that an organization was needed to centralize and coordinate all radio propagation research of the Federal Government. The Central Radio Propagation Laboratory of the National Bureau of Standards was established to assume this responsibility.

CRPL is divided into three laboratories: ionospheric research, systems research, and measurement standards. The coordinated program of radio propagation research at frequencies above approximately 50 megacycles is centered in the systems research laboratory. The chief aim of this group is to help solve the problems associated with the allocation of frequencies for television, FM, aircraft communication, traffic control, and navigational systems.

At frequencies above about 50 megacycles, an entirely new set of conditions influence radio wave propagation. In the standard broadcast band (550-1,600 kc) electrical characteristics of the soil and conditions of the lower ionospheric regions play the dominant roles in determining the strength of the signal and the distance to which it will be propagated. In the international high-frequency bands below 50 megacycles the condition of the ionosphere alone is the dominant factor. At frequencies above about 50 megacycles, however, climate, meteorological conditions in the troposphere, and terrain irregularities play the principal roles, and very different climatological and terrain situations influence the propagation characteristics of radio waves.

Propagation data which will have useful application under the various conditions of climate and terrain encountered throughout the world must be obtained from a variety of geographical locations and over extended periods of time. In general, it has been found desirable to record radio field strengths over particular paths for periods of one year or greater in order to determine the seasonal factors and the year-to-year variability of field strength. Mobile measurements are desirable to study terrain factors affecting the propagation. In most cases it has been found neces-



A nation-wide network (see map) of NBS field stations, commercial radio installations, engineering consultants, and FCC monitor stations provides experimental data for the VHF-UHF field strength recording program of NBS. Transmitting antenna (left) at NBS station atop Cheyenne Mt., Colo., radiates 100- and 192.8-Mc signals from top arrays; 1046-Mc signals from semipyramidal horn at base. A typical receiver installation (right) is located at Kendrick, Colo.



sary to employ statistical sampling techniques, which are becoming more and more important in the study of all types of propagation factors varying with time and space. Propagation measuring equipment which must be used at these higher frequencies is generally not available and equipment development is also involved.

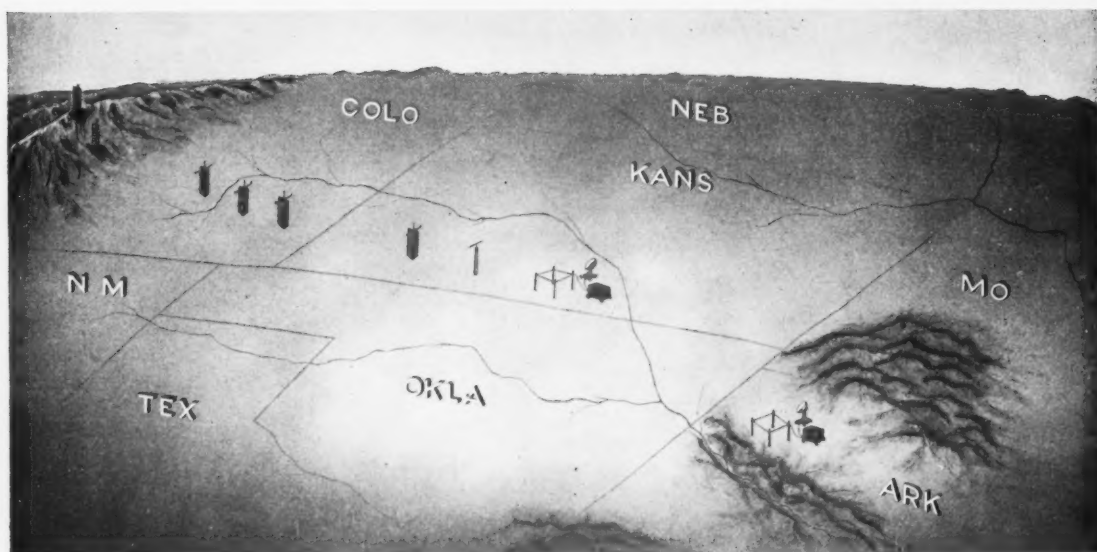
The regular tropospheric propagation research program of NBS is designed to investigate the propagation phenomena from both the theoretical and experimental point of view. The work is divided into four main projects: (1) terrain effects on propagation, (2) tropospheric effects on propagation, (3) tropospheric propagation measurement and analysis, and (4) tropospheric and irregular terrain propagation experiments at Cheyenne Mountain in the 100 to 1,000-Mc region. A large part of the experimental measurements program utilizes existing Government and commercial transmitting stations, the facilities of universities, established engineering consultants, and monitoring stations of the Federal Communications Commission.

One phase of the work has been the study of air-to-air and air-to-ground VHF and UHF communication systems for the Departments of the Air Force and the Navy. Another phase has been the study of UHF propagational effects, directed toward air navigation and traffic control systems, for the Air Navigation

Development Board. Still another phase has been the establishment of a number of field strength measuring stations throughout the United States to obtain data on the effects of climate, terrain, antenna height, and frequency on the propagation of VHF and UHF radio signals at distances well beyond the horizon. Earlier studies of this type yielded information needed by the FCC in connection with their allocation of FM and television broadcast stations. The experimental research program established at the NBS field station on Cheyenne Mountain, Colorado, is designed to supply information on the radio propagation conditions experienced by an aircraft in flight. The investigation involves the study of signals received at distances far beyond the horizon—much farther than predicted by existing theories.

Terrain Effects on Propagation

Several NBS mobile units are operating in various parts of the United States gathering field-strength data from a number of transmitting stations under varied receiving and transmitting conditions. One unit, equipped with sensitive field-strength recorders and calibrated antennas, was used in a series of investigations of several TV and FM stations in the Baltimore-



Pictorial representation of the Bureau's installation at Cheyenne Mountain, Colo. Upper left: Two transmitter sites are located at 9,000 and at 7,000 feet above sea level. The summit location transmits signals at frequencies of 100, 192.8, and 1046 Mc; the signals from the lower site are 100 and 192.8 Mc. The radio energy is beamed eastward across the plains of Colorado and Kansas, and has been received by mobile receiver units 600 miles from the transmitter. Permanent receiver installations are located near Kendrick, Karval, and Haswell in Colorado, and Garden City and Medicine Lodge in Kansas. Mobile units operate near Anthony, Kansas, and Fayetteville, Ark.

Washington area and of experimental transmitters at Fort Dix, N. J.

In the first of these investigations, the mobile unit operated approximately midway between two television stations transmitting on adjacent channels and measured the amount of interference presented by each station. The variations in field strength were also measured as a function of receiving-antenna height in which a calibrated antenna could be accurately set at levels between 10 and 30 feet. The interference characteristics of TV stations operating on near or adjacent channels but within the same general transmitting area were also investigated. An accompanying study was conducted on the effects of frequency on received signal strength by investigating the performance of an installation transmitting two frequencies from the same antenna, as is being done by stations WRC-FM and WNBW in Washington.

This same mobile unit was used to record the field strengths of the Army Signal Corps experimental transmitters operating at 49, 141.75, 239, and 460 Mc. Ground plane antennas were constructed for each of the operating frequencies, and these units and the receivers were all calibrated at the National Bureau of Standards prior to their being used. Recordings were made continuously while driving along several approximately circular routes around the transmitters. At some locations along the route, the transmitting antenna could be seen; at others, the antenna was obscured by obstacles nearby.

An investigation of the effects of terrain on the directivity of high gain antennas has been made using VHF

and UHF transmissions of the United States Army Signal Corps. The resulting data show many variations in the apparent radiation patterns of the transmitting antennas. In many instances the direction at which the maximum signal was received was far from the direction of maximum for which the transmitting antennas were oriented. A directive antenna operated in the mobile unit showed many variations in pattern but seldom did the direction of maximum reception differ appreciably from the direction toward the transmitter. The exceptions occurred only when a large number of obstacles were present to deflect, absorb, and reflect the transmitted signal.

Ground conductivity plays a dominant role in the transmission of low frequencies in the 550-1,600 kilocycle region. Radio broadcasters have long realized that a signal generated in Louisiana will be received more readily at long distances than one originating along the rock-bound coast of New England. The FCC has provided NBS with a large quantity of ground conductivity measurements that have been made by the various consulting engineers in connection with directional antenna proof-of-performance surveys. In addition, the Department of Agriculture has submitted a series of maps dividing the soils of the United States into approximately 250 classifications.

The purpose of the NBS analysis is to correlate the ground conductivity measurements with soil types. Although the program is only partially completed, there is an indication that a better method of presenting Nation-wide ground conductivity characteristics is to assign a most probable conductivity to each of the 250

Mobile field strength recording unit used to obtain data from a number of transmitting stations under varied receiving and transmitting conditions. The van is equipped with a 50-foot, calibrated, power-driven mast antenna and sensitive field-strength recorders. The mobile unit was used in a series of investigations of several TV and FM stations in the Baltimore-Washington area and of experimental transmitters at Fort Dix, N. J. The building in the background houses the Tropospheric Research Laboratory at Boulder, Colo.

types, together with a possible range of values that might occur in each soil group.

Tropospheric Effects on Propagation

In the middle of 1950, the Department of Defense requested a rapid compilation and analysis of tropospheric information to assist in determining the performance, interference, and interception characteristics of military systems operating in the VHF and UHF portion of the spectrum. The Bureau supplemented its Nation-wide recording program for determining the required propagation characteristics with additional recordings from the University of Washington, the University of Pennsylvania, and the Federal Communications Commission monitoring stations. Thus a more adequate coverage of different types of climate and terrain was achieved. Field strength recordings at UHF have been initiated at Quincy, Illinois. Radio signals from the Collins Radio Company 418 Mc, high power, resnatron transmitter in Cedar Rapids, Iowa, are directed toward receiving antennas at various elevations on the 750-foot tower of WTAD-FM in Quincy, a path approximately 135 miles long.

The link between meteorological conditions and tropospheric propagation is the radio index of refraction of the atmosphere. Refractive indices are computed from data obtained by radiosonde (free balloon ascent), wiresonde (captive balloons), or from instruments carried in aircraft. An analysis of meteorological data for elevations greater than 50,000 feet, collected by the U. S. Weather Bureau, is now in progress at NBS. These data are the accumulation of

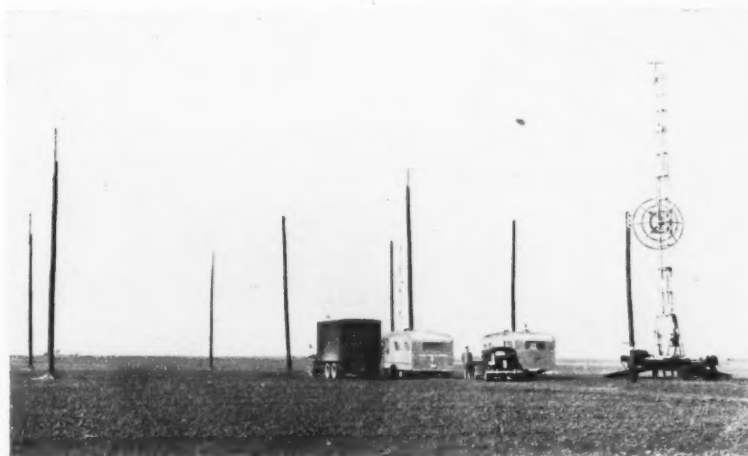


reports collected for many years by some 50 U. S. Weather Bureau stations. It is hoped that the analysis will lead to a better understanding of the variations of tropospheric propagation with respect to the weather over long distances in different parts of the country.

An analysis of the measurements made at the National Bureau of Standards and at other laboratories performing the same type of investigations has indicated that the conventional constants in the expression for the refractive index of moist air should be revised. Several laboratories have arrived at this conclusion independently with the result that there are at least four different sets of constants now in current use. A thorough study by members of the Bureau staff of the literature and of NBS data has resulted in the determination of the "best" values of the constants in the statistical sense.

Tropospheric Propagation Measurement and Analysis

Data is funneled to NBS from the cooperating commercial transmitting stations, university facilities,



Mobile receiver-installation. Units such as these travel the length of a 600-mile path between Colorado Springs, Colo., and Fayetteville, Ark., recording the field-strength of signals transmitted from Cheyenne Mountain. The three vehicles included in the unit are a power supply van (left), receiving and recording equipment van (middle), and sleeping quarters (right). The telephone poles support the lower frequency antenna system (100 and 192.8 Mc) and the trailer-mounted antenna is used for the 1,046-Mc operations.

engineering consultants, and the Federal Communications Commission monitoring stations. These data need to be analyzed in the light of theoretical considerations for the purpose of developing more complete and accurate physical, mathematical, and statistical methods of describing tropospheric propagation over irregular terrain.

The prediction of expected tropospheric field strengths versus distance and transmitting and receiving antenna heights for any time of day and season and any region of the world requires three conditions: (1) an adequate description of the behavior of available data; (2) an analysis of this behavior in the light of theories of propagation; (3) a close correlation of variability in time with relevant meteorological information. The last two requirements involve not only general comparative statistics but also refined methods of analysis. The methods differ in the mathematical form that a trend is assumed to follow. The most successful method used thus far by the NBS group for the above application is a type of harmonic analysis that assumes the trend to be a trigonometric polynomial, that is, the sum of harmonic sine and cosine components with a fundamental period of a year. In using the harmonic analysis method, the coefficients of the trigonometric terms are resolved as well as an expression for the trend. This type of analysis involves the summation of thousands of numbers. As a means for shortening the many hours of machine computations that calculations of this type require, the design of a special electronic computer to handle only this type of problem is now being considered by members of the NBS staff.

Cheyenne Mountain Experiments

Progress has been made in the air navigation field toward the development of a unified system of air communication, navigation, and traffic control. This work is centralized in the Air Navigation and Development Board, administratively under the Civil Aeronautics

Administration, which is concerned with a unified system of air navigation to operate in the frequency range of 960–1,600 Mc. Very little radio propagation information exists for this frequency range.

The NBS tropospheric propagation research facilities at Cheyenne Mountain have been established to determine the necessary propagation factors for the effective allocation and use of these frequencies. The program has included the development, installation, and operation of a system for studying propagation factors at 1,000 Mc. The system utilizes a transmitter that gives the highest continuous power output in this frequency range of any transmitter ever developed in this country. The operating frequency is 1,046 Mc; a moderately directive, semipyramidal horn antenna radiates 1.6 megawatts of effective power. The transmitter is now operating on a regular schedule for the systematic collection of tropospheric propagation data. High power is used so that reliable propagation data can be obtained in the distant interference region of the transmitter. This development marks a new departure in propagation research, which in the past has followed rather than preceded the use of a new portion of the frequency spectrum.

The program of NBS tropospheric propagation research has grown steadily through the past few years, reflecting the various requirements of the many government agencies and other users. Actually, of course, this program is entirely inadequate to provide a general solution to the tropospheric propagation problems in the frequency bands now usable above 50 Mc. A concerted effort is being made only to meet the urgent requirements arising from the need for solutions to specific allocation problems in accordance with the recommendation of the President's Communications Policy Board. It is anticipated that further requirements will be forthcoming as the result of the development of other systems and these will require more intensive research efforts. Current NBS activities involve considerable basic research which will be useful in future systems involving radio propagation.

New Miniature Test Probes

TWO TYPES of miniature test probes recently devised at the Bureau are being used to speed development and testing in the NBS radar miniaturization laboratory. Light and compact, the probes are designed to cling to the test point without danger of contacting adjacent leads. Intended particularly for use with miniaturized electronic equipment, the new probes offer possible advantages for use with conventional-sized devices as well.

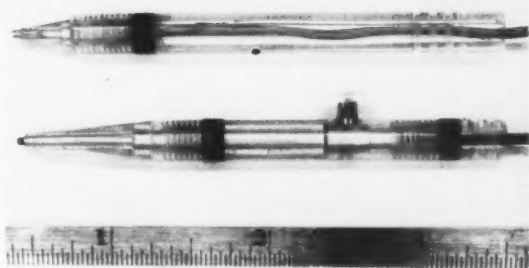
One of the probes is a push-on type, with a very small tapered jaw that is simply pressed onto the wire under test. The jaw is of hardened beryllium copper, silver-plated for good electrical contact. It grips the wire with a slight spring action until sufficient pull is exerted to remove it. The jaw screws into an insulat-

ing handle, made of lucite or of material like fiber having greater mechanical strength and heat resistance. The handles now being used at NBS are $\frac{1}{4}$ inch in diameter and $3\frac{1}{2}$ inches long. Color-coding of transparent-handled probes is accomplished by using colored lead wires, while colored bands are placed in grooves in the fiber-handled probes. Only about $\frac{1}{16}$ inch of the metal jaw protrudes from the insulating handle, so that the danger of shorting to nearby components is minimized.

The other probe is a lock-on type, designed so that it cannot be removed from the wire until a release button on the side of the probe is pressed. A small hook mechanism at the end of the probe remains open only while the button is pressed, and tightens on the

Two miniature test probes recently developed at the Bureau are particularly useful in servicing miniature electronic equipment. A push-on type (top) is designed to grip the wire on which it is pressed, until pulled away. A lock-on probe (bottom) has a small hook in its tip, controlled by a button on the side of the probe; this probe cannot be removed from the wire until the release button is pressed.

wire when the button is released. In other respects, including size, the lock-on probe is similar to the push-on model. Like the push-on model, the lock-on probe is designed to accommodate wires varying considerably in diameter. Although several moving parts are required, manufacture of the lock-on probe is straightforward.



Electron-Optical Bench

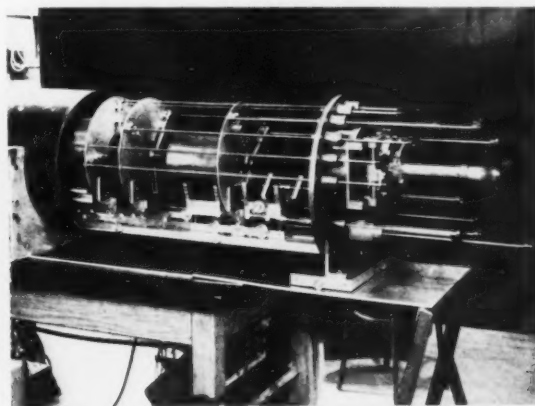
A VERSATILE electron-optical bench has recently been constructed at the Bureau for the extensive study of electron-optical elements. Carriages for magnetic lenses, mirrors, or prisms and holders for aperture objectives, and meshes are arranged appropriately in a vacuum chamber. External positioning controls are also provided, offering three degrees of freedom for each element. The bench was developed by the NBS electron physics laboratory under the direction of Dr. L. Marton. It has become an integral part of a program devoted to the investigation of extremely small electric and magnetic fields in spaces that have heretofore been inaccessible to conventional types of measurement.

Electron-optical methods have been successfully applied to the measurement of magnetic fields around magnetized recording wires, the fringe fields issuing from ferro-magnetic domains, and the space-charge

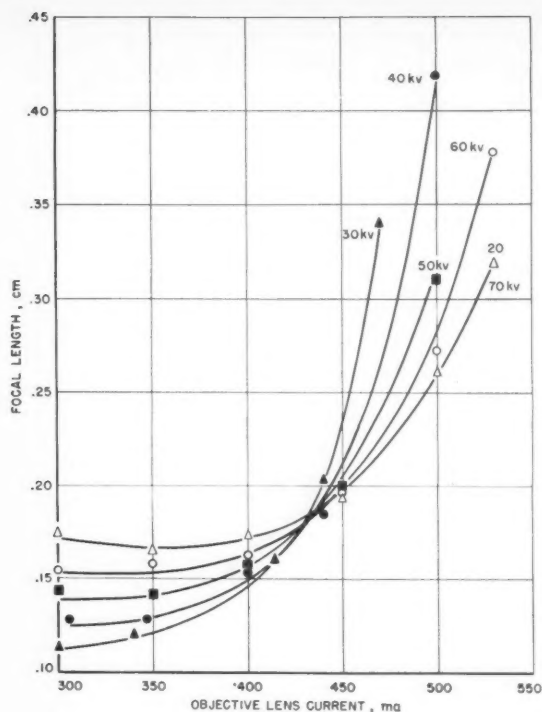
distribution of a d-c cut-off magnetron. Besides being able to measure these fields, the NBS electron-optical bench can be adapted for electron-optical field mapping, as an electron microscope, electron diffraction camera, or any instrument having closely related characteristics.

For simplicity of construction and protection against X-rays, the bench is made with an all-metal cylindrical vacuum chamber. It is designed to satisfy the following requirements: (1) To accommodate three carriages for lenses with maximum diameters of 7 in. and four holders for apertures, test objects, and meshes (for use in field mapping); (2) to permit a movement of 10 in. along the axis of the bench and one-half inch radially for each component of the system without breaking the vacuum.

One end plate of the electron-optical bench supports the electron gun and is permanently fixed to the cylinder with bolts and a rubber gasket vacuum seal. A heavy



Left: the NBS electron-optical bench being used as an electron diffraction camera. The electron-optical lens, which produces diffraction, is connected to the front of the bench, and a cathode ray tube is inserted to permit visual observation of the transmission pattern. The high-voltage power supply (background) is controlled from the panel immediately to the left of the bench. **Right:** the electron-optical bench removed from the vacuum chamber. The bench is designed so that it may be rolled out of the chamber on roller-bearing trucks onto a removable extension table (lower right). Mounted on the bench are the lenses (open cylinders) and the test objects or aperture mounts (disks). The positions of these elements are controlled by means of the rods passing through the face plate at right.



dural plate serves as the bed for the electron-optical system and is provided with wheels so that the whole system may be rolled in or out of the chamber. The face plate is sealed against the end of the cylinder by a neoprene gasket compressed by atmospheric pressure. A fluorescent screen ($5\frac{7}{8}$ in. in diameter and $\frac{7}{8}$ in. thick) and "Wilson seals"—through which pass the connecting shafts that control the motion of the elements—are inserted into the face plate.

Radial adjustment of each element is obtained by properly combining controlled vertical and horizontal motions of the lens carriage and holders, achieved by worm and gear arrangements. The axial position of the carriage is controlled by pushing or pulling the connecting shafts through the Wilson seals. Radial motion is constrained by elastic deformation of phosphor bronze strips instead of sliding surfaces.

Current leads for the electron lenses enter the vacuum chamber through metal-glass seals in the face plate; the conductors are insulated by ceramic beads, which produce fewer outgassing difficulties than would result if rubber or plastic insulation were used. The lenses available for use in the bench were originally designed for use with the coils out in the atmosphere rather than in a vacuum chamber. Because the coils release large quantities of gas to the vacuum system, a rapid pumping system is needed to maintain a working vacuum under these unfavorable conditions. A large fore-pump brings the pressure in the chamber down to about 10 microns of mercury in about 5 minutes. A 4-in. oil diffusion pump is used to maintain an operating

Graph showing the variation in the focal length of an objective lens current for different accelerating voltages on the NBS electron-optical bench. The focal-length measurements were made with the condenser lens, a mesh object, and the objective lens in the system. The maximum error in determining the focal length was about ± 0.01 cm.

pressure of about 3×10^{-2} micron of mercury, measured by an ion gauge installed near the electron gun.

A conventional 100-kilovolt X-ray power supply, consisting of a voltage doubler circuit and two 50-kilovolt rectifier tubes, is used as the high-voltage supply; the normal operating range is between 20 and 70 kilovolts. The filaments are heated by a high-frequency power supply. Lens currents are supplied from separate power supplies that yield currents up to 500 milliamperes and have a stability of better than 1 part in 10,000. The grid bias for the 100-kv electron gun is obtained from a potentiometer connected across three 45-volt batteries in series.

A large proportion of the measurements made with an electron-optical bench are empirical evaluations of electron-optical systems. In such work the results of one measurement often determine the conditions for the next. Hence, a direct method of measuring the image is preferred because it is faster, less involved and less expensive than the photographic method of recording results. A specially built low-power traveling telescope, reading to 0.0001 in. and with total travel of $2\frac{3}{4}$ in., is used to measure the image on the fluorescent screen. Because the NBS bench is designed for use with magnetic lenses, which cause rotation of the image, the measuring telescope is mounted on a turntable. It can be moved a distance of 3 in. either horizontally or vertically so as to keep its center of rotation concentric with that of the image. Thus, all measurements are radial. Variable-intensity side illumination of the reticule reduces the difficulty in seeing the cross hair when the intensity of the pattern on the screen is low.

One of the initial measurements made with the NBS electron-optical bench was that of the focal length of a magnetic lens. The lens was of the condenser type, and the system also included a mesh object and a 10,000-turn objective lens. Diffuse illumination of the object, obtained by applying a thin formvar film and a thin conducting aluminum coating on the source side of the mesh, was used to make the focusing critical. The image distance was held constant throughout the measurements. For a particular accelerating voltage, the object position was adjusted to give approximate focus for different objective currents, the final focus being made by a small adjustment of the current. The focal length was computed from the equation $f = q/(M+1)$, where q is the image distance and M is the magnification.

A graph of focal length vs. objective lens currents for different accelerating voltages shows that the maximum error in determining the focal length was about ± 0.01 cm. The major limitation on accuracy seems to be the difficulty in setting the cross hair on a point

of the mesh image. Improvement of the cross-hair design and development of greater skill in setting may reduce the error due to this source to less than 1 percent. The precision with which the image can be focused imposes another limitation. The objective lens and pole piece used in the initial measurement permits the image to be apparently in perfect focus over a current range of about 6 ma. The center of this range can be located within about ± 2 ma. It is believed that this cannot be much improved without

elaborate additions to the equipment. Errors due to small inaccuracies in alignment and to hysteresis are believed to be negligible compared to the two limiting factors.

For further technical details, see Electron-optical bench, L. Marton et al., NBS J. Research 47, No. 6, 461 (RP 2273). See also Electron-optical shadow method, NBS Tech. News Bul., 33, No. 9, 106 (Sept. 1949) and Electron-optical mapping of the space-charge filed in a magnetron, NBS Tech. News Bul., 34, No. 5, 57 (May 1950).

Purification of Zirconium

A rapid, convenient method for freeing zirconium of common impurities has recently been developed by W. S. Clabaugh and Raleigh Gilchrist of NBS. The zirconium is obtained as the sulfate in good yield and very high purity. The method also provides a new technique for preparing zirconium oxide which may find application in commercial preparation of the metal.

Because of the complex nature of zirconium salts and the incomplete knowledge of their chemistry, the purification of zirconium has until now been a difficult matter. As a result, most laboratories have had to use zirconium in whatever purity they were able to purchase it. The NBS method, however, makes it possible for each laboratory to obtain zirconium free of all common impurities except hafnium.

It was found at the Bureau that when concentrated sulfuric acid is poured into a fairly concentrated aqueous solution of zirconium sulfate or chloride, a dense white crystalline precipitate of $Zr(SO_4)_2 \cdot 4H_2O$ is formed. This compound is soluble in an equal weight of water. By dissolving the precipitate in water and reprecipitating the hydrated sulfate several times, zirconium sulfate of high purity is obtained.

For best results, one volume of concentrated sulfuric acid is added to two volumes of concentrated zirconium solution. The zirconium sulfate precipitate is caught on a sintered glass filter of medium or coarse porosity and washed with acid and acetone solutions. A small amount of hydrochloric acid is added during recrystallization to prevent the precipitation of any iron that may be present as an impurity.

The solution used to wash the precipitate consists

of 75 volumes of water, 40 volumes of concentrated sulfuric acid, and 5 volumes of concentrated hydrochloric acid. After several washings with this mixed acid, three washings with acetone are recommended. Alcohol, however, cannot be used for this final washing because in some way it causes interference in subsequent recrystallizations, probably through complex formation.

In a recent test run at NBS, the method was applied to 1.135 grams of commercial zirconium chloride containing about 0.3 percent of iron and 2.7 percent of hafnium. The zirconium chloride was dissolved in water to make 2.1 liters of solution, 1 liter of sulfuric acid was added, and the resulting product was recrystallized five times. A 70-percent yield (1.212 grams of zirconium sulfate) of high purity was obtained. Chemical analysis of the final product showed that it contained less than 0.1 part per million of iron and less than 0.1 part per million of copper. Spectrochemical methods gave a silver content of less than 1 part per million and less than 10 parts per million each of calcium, magnesium, sodium, and silicon. No other elements except hafnium were detected spectrochemically. A trace (about 0.01 percent) of chloride ion was present, but this could be removed merely by recrystallizing the zirconium sulfate from water containing no hydrochloric acid after the iron had been eliminated.

The final product formed upon ignition of the hydrated sulfate was examined by X-ray diffraction and found to be the monoclinic form of ZrO_2 . Thus, the NBS procedure also provides a convenient method for obtaining the oxide.

Electronic Components Symposium

The National Bureau of Standards participated actively in the Symposium on Progress in Quality Electronic Components that was held in Washington on May 5, 6, and 7. A sequel to a similar event held in 1950, the 1952 Symposium was attended by more than 1,100 engineers and technical experts from the electronics industry, the military services, and research laboratories. The Symposium was sponsored jointly by the American Institute of Electrical Engineers, the Institute of Radio Engineers, and the Radio-Television Manufacturers Association, with the cooperation of Department of Defense organizations and NBS. J. G. Reid, Jr., chief of the NBS Electronics Division, served as chairman of

the Symposium Committee, and NBS Director Dr. A. V. Astin presided over the first session.

More than 40 technical papers were presented in a series of sessions devoted to such categories as Basic Materials, Capacitors and Inductors, Resistors, Electron Tubes, Transistors, Advances in Miniaturization, and Aspects of Reliability. B. L. Davis and J. H. Muncy, both of the NBS Engineering Electronics Section, gave papers on "Adhesive Tape Resistors" and "Electronic Failure Prediction," respectively. Proceedings of the Symposium have been published and are available from the RTMA in Washington at \$5 a copy.

Publications of the National Bureau of Standards

PERIODICALS

- Journal of Research of the National Bureau of Standards, volume **49**, number 1, July 1952 (RP2336 to RP2341, incl.)
 Technical News Bulletin, volume **36**, number 7, July 1952, 10 cents.
 CRPL-195. Basic Radio Propagation Predictions for October 1952. Three months in advance. Issued July 1952. 10 cents.

RESEARCH PAPERS

Reprints from *Journal of Research*, volume **48**, No. 6, June 1952

- RP2329. Effect of strain-temperature history on the flow and fracture of ingot iron at low temperatures. Glenn W. Geil and Nesbit L. Carville. 10 cents.
 RP2330. Gradient methods in the solution of systems of linear equations. Marvin L. Stein. 10 cents.
 RP2331. An alkaline solution of potassium chromate as a transmittancy standard in the ultraviolet. G. W. Haupt. 10 cents.
 RP2332. Two applications of group characters to the solution of boundary-value problems. E. Stiefel. 5 cents.
 RP2333. Heterogeneous equilibria at the glass-electrode-solution interface. Donald Hubbard and Richard G. Goldman. 10 cents.
 RP2334. Preparation of D-mannitol-C¹⁴ and its conversion to D-fructose-1-(and 6)-C¹⁴ by acetobacter suboxydans. Horace S. Isbell and J. V. Karabinos. 5 cents.
 RP2335. Analysis of goniophotometric reflection curves. Isadore Nimeroff. 10 cents.

CIRCULARS

- C499. (Supplement No. 3). Nuclear data (January 1951 to July 1951). (Last of three supplements that will be mailed automatically to purchasers of Circular 499.) Price, Circular 499 and three supplements, \$4.25.

APPLIED MATHEMATICS SERIES

- AMS13. Tables for the analysis of beta spectra. 35 cents.
 AMS17. Tables of coulomb wave functions. Volume 1. \$2.00.

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- The variation of intensity of fast cosmic-ray neutrons with altitude. L. F. Curtiss and P. S. Gill. Phys. Rev. (57 East Fifty-fifth Street, New York 22, N. Y.) **85**, No. 2, 309 (January 15, 1952).
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Publications for which a price is indicated are available only from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. (Foreign postage, one-third additional). Reprints from outside journals are not available from the National Bureau of Standards, but can often be obtained from the publishers.

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